भारतीय मानक
Indian Standard

IS 17378 : 2021

फ्लेम एटॉमिक एब्ज़ोर्प्शन स्पेक्ट्रोस्कोपिक पद्धति द्वारा कोयले और कोक की राख में प्रमुख घटक ज्ञात करना

Determination of Major Elements in Ash from Coal and Coke by Flame Atomic Absorption Spectroscopic Method

ICS 73.040, 75.160.10

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Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07

FOREWORD

This Indian Standard was adopted by Bureau of Indian Standards after the draft finalized by the Solid Mineral Fuels and Solid Biofuels Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Compositional analysis of ash from coal/coke is often useful in total description of the quality of coal/coke. Knowledge of ash composition is also useful in predicting the behavior of ashes and slags in combustion chambers. It also aids in the better utilization of ash by-products as their properties may also depend on the chemical composition of the ash.

Note that the chemical composition of laboratory-prepared coal/coke ash may not exactly represent the composition of mineral matter in the coal/coke or the composition of fly ash and slag resulting from commercial-scale burning of the coal.

Ash as determined is not the same as the original mineral matter associated with the coal/coke, because changes take place during incineration, namely the loss of combined water or carbon dioxide from carbonates, etc. Recommended formulae for the calculation of mineral matter in coal from ash are given under **6** of IS 4311 : 1967 'Method for the determination of mineral matter in coal'.

In formulation of this standard, due weightage has been given to the international co-ordination among the standards and practices prevailing in different countries in addition to the practices in India and considerable assistance derived from the following international standards:

ISO 23380 : 2013	Selection of methods for the determination of trace elements in coal
ASTM D3682-13	Standard test method for major and minor elements in combustion residues from coal utilization processes

The composition of the Committee responsible for the formulation of this Indian Standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).'

Indian Standard

DETERMINATION OF MAJOR ELEMENTS IN ASH FROM COAL AND COKE BY FLAME ATOMIC ABSORPTION SPECTROSCOPIC METHOD

1 SCOPE

This standard prescribes the methods for sample preparation, dissolution and flame atomic absorption spectroscopic method for the determination of common major elements in ash from coal or coke.

2 REFERENCES

The standard listed below contains provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard listed below.

IS No.	*Title*
1350 (Part 1) : 1984	Methods of test for coal and coke: Part 1 Proximate analysis (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
264 : 2005	Nitric acid — Specification (*third revision*)

3 TERMINOLOGY

For the purpose of this standard, the following definition shall apply.

3.1 Ash — Inorganic residue left when powdered coal or coke is incinerated in air to constant mass under specified conditions.

4 PRINCIPLE

Ash sample prepared from coal or coke is fused with lithium tetra borate ($Li_2B_4O_7$) followed by a final dissolution of the melt in mineral acids (dilute hydrochloric acid or dilute nitric acid). The solution is analyzed by atomic absorption/ emission for applicable elements.

5 REAGENTS

5.1 Quality of Reagents

Reagents used in preparation of standard stock solution for each element shall be pure and free from elements to be determined. Stock solutions of 1 000 ppm and 100 ppm of metal ions shall be stored in polythene/ PTFE bottles with leak-proof screw caps and labeled with the date of preparation. The stock solution shall not be used any longer than twelve months. Working and calibration standards are prepared by step dilution of stock solution each day before analysis. The lithium tetra borate and lanthanum chloride reagents in particular shall be examined for alkali and alkaline earth contamination.

5.2 Quality of Water — Unless specified otherwise, distilled water [*see* IS 1070] shall be used in tests.

5.3 Lithium Tetra Borate ($Li_2B_4O_7$) — Powder.

5.4 Solutions

5.4.1 *Concentrated Hydrochloric Acid* — Specific gravity 1.18 [*see* IS 265].

5.4.2 *Concentrated Nitric Acid* — Specific gravity 1.42 [*see* IS 264].

5.4.3 *Solvent Acid* — Dilute 50 ml of concentrated hydrochloric acid (**5.4.1**) or 50 ml of concentrated nitric acid (**5.4.2**) to 1 000 ml. Any of these acid solutions may be used, but whichever is chosen shall be used throughout the subsequent solution preparations.

5.4.4 *Lanthanum Chloride Solution* — 175 g/l lanthanum chloride ($LaCl_3$) or equivalent 10 percent lanthanum.

5.5 Stock Solutions

5.5.1 *Aluminum Stock Solution* **—** 1 000 ppm aluminum.

5.5.2 *Calcium Stock Solution* **—** 1 000 ppm calcium.

5.5.3 *Iron Stock Solution* **—** 1 000 ppm iron.

5.5.4 *Magnesium Stock Solution* **—** 1 000 ppm magnesium.

5.5.5 *Potassium Stock Solution* — 1 000 ppm potassium.

5.5.6 *Silicon Stock Solution* — 200 ppm silicon.

NOTE — A standard stock solution may be prepared by fusing 0.1070 g of reignited spectrographic grade silica (SiO_2) with 1 g of lithium tetra borate, dissolving it in solvent acid, and diluting to 250 ml as described for sample preparation in **7.2.1** and **7.2.2**. This solution is 200 ppm silicon. The standard sample(s) should have composition(s) similar to the unknown.

5.5.7 *Sodium Stock Solution* — 1 000 ppm sodium.

5.5.8 *Titanium Stock Solution* — 1 000 ppm titanium.

6 APPARATUS

6.1 Atomic Absorption Spectrometer

The atomic absorption spectrometer shall have in the absorption mode, a deuterium (D_2) arc background corrector or other comparable simultaneous background correction system.

6.2 Ashing Furnace — With an adequate air circulation and capable of being raised to 815 ± 10°C.

6.3 Platinum Dish — 35 to 85 ml capacity.

NOTE — To minimize the potential of contamination, the platinum ware must be cleaned by boiling in solvent acid and rinsing thoroughly with reagent-grade water. After this initial cleaning, the platinum ware must be handled with clean tongs and protected from further contamination from table tops, and so forth. All glassware used in analyses must be cleaned and protected from contamination.

6.4 Stirring Hotplate and Bars — Operating temperature of 200 ± 5°C.

6.5 Balance — An analytical balance capable of weighing upto 200 g with least count 0.1 mg.

7 ASH SAMPLE AND SAMPLE SOLUTION PREPARATION

7.1 Preparation of Ash Sample

7.1.1 Coal/coke samples which are received in the laboratory already ground to pass IS sieve 20 (212 microns) shall be re-sieved to verify that all the material passes the sieve, and then the sample is air-dried for 24 h and mixed and bottled.

NOTE — Should a sample of coal or coke be transported to any distance between the point of preparation and the laboratory under conditions in which it may be shaken down and may cause segregation to any degree, the entire contents of the bottle shall be re-mixed before analytical work is started. The use of mechanical devices is recommended.

7.1.2 Thin layers of coal or coke spread in the uncovered dish is introduced into the ashing furnace at room temperature, temperature is raised to 500°C in 30 min and to 815 ± 10°C further in 30 to 60 min and maintained at this temperature for 60 min until the residue is constant in mass as specified in **8** of IS 1350 (Part 1).

NOTES:

1 The conditions for the determination of the ash content of coal or coke as specified in **8** of IS 1350 (Part 1) ensure the reproducibility of results in different laboratories.

2 Under these conditions of preparation, the ash shall be free from carbonates and all small traces of sulphide, but all the iron compounds should be fully oxidized. Previously ashed materials are ignited to constant weight under standard conditions.

7.2 Sample Solution Preparation

7.2.1 The solutions in the proportions described below are for typical ash samples as represented by Indian coals of sub bituminous and bituminous rank and cokes. Therefore, stronger or weaker dilutions may be required to establish suitable concentrations for those elements of varying percentages outside the range of the typical sample. Each analyst must determine the sensitivity and linear range of calibration of their equipment and choose concentration ranges for standards compatible with the samples and instrument specific to their own work.

7.2.2 Weigh 0.1 ± 0.0002 g of the ash sample as prepared in **7.1** into a platinum dish (**6.3**) and add 0.5 g of lithium tetra borate. Mix them well, then add an additional 0.5 g of lithium tetra borate to cover the mixture. Place the dish in a clean silica or refractory tray and place in a muffle furnace preheated to 1 000°C, 15 min at 1 000°C is sufficient to fuse the mixture completely. Remove the tray and dish and cool to room temperature. Carefully rinse the bottom and outside of the platinum dish to remove possible contamination, then place it in a clean 250 or 400 ml beaker. Place a clean TFE fluorocarbon-coated stirring magnet inside the dish, add 150 ml of solvent acid (**5.4.3**) to the beaker and dish, and place immediately on the stirring hotplate. Heat the solution to just below boiling temperature and maintain for not more than 30 min with constant stirring. This time and temperature are sufficient to completely dissolve the melt. If stirring is not maintained constantly, some of the ash constituents are apt to precipitate and the analysis must be repeated.

7.2.3 Remove the beaker from the hotplate and permit to cool to room temperature. Quantitatively transfer the solution to a 200 ml volumetric flask, wash the platinum dish and beaker with small amounts of solvent acid and dilute to the 200 ml mark with the solvent acid. This solution is 500 ppm with respect to the total sample and contains 5 g/l of lithium tetra borate solution.

7.2.4 *Solution 1* — Pipet 20 ml of the sample solution prepared in **7.2.3** into a 50 ml volumetric flask and dilute to volume with solvent acid. This solution is 200 ppm with respect to the total sample and contains 2 g/l of lithium tetra borate solution.

7.2.5 *Solution 2* — Pipet 10 ml of the sample solution prepared in **7.2.3** into a 100 ml volumetric flask, add 10 ml of lanthanum solution (**5.4.4**) and dilute to volume with solvent acid. This solution is 50 ppm with respect to the total sample and contains 0.5 g/l of lithium tetra borate solution.

NOTE — Lanthanum was included in the solution as a release agent in those determinations that may require it (for example, calcium). Alternative measures, such as the use of nitrous oxide flame, are permissible.

8 PROCEDURE

8.1.1 Calculations used in subsequent clauses are developed from the following general formula for percent concentration of element oxide, E, in moisture-free coal ash:

$$E= [(C - B)/(A - B)] \times (N/M) \times F \times 100 \quad \ldots\ldots (1)$$

Where,

A = Absorbance of standard;

B = Absorbance of blank;

C = Absorbance of sample solution;

N = Element in standard, ppm;

M = Sample of solution, ppm; and

F = Conversion from element to oxide.

8.1.2 Concentrations of element oxides in the ash may be converted to the air-dried coal basis using the following expression:

$$C = (A \times B/100) \quad \ldots\ldots (2)$$

Where,

C = Element oxide, in percent, in air-dried coal;

A = Element oxide, in percent, in ash; and

B = Ash, in percent, as determined in **8** of IS 1350 (Part 1).

8.2 Silicon Dioxide (SiO_2)

8.2.1 *Preparation of Standard*

Prepare a series of standards by combining the volumes of the silicon stock solution (**5.5.6**) as given at Table 1 with 1 ml of the aluminum stock solution (**5.5.1**) and diluting to 100 ml with the blank solution (**8.2.2**). Only those standards need be prepared that bracket the expected SiO_2 concentrations in the samples.

8.2.2 *Blank Solution*

Prepare 2 g/l of lithium tetraborate solution in solvent acid.

Table 1 Silicon Stock Solutions for Preparation of Standard Silicon Solution

(*Clause* 8.2.1)

Sl No. (1)	Silicon Stock Solution (5.5.6), ml (2)	Silicon, ppm (3)	Equivalent SiO_2, Percent (4)
i)	25	50	53.5
ii)	20	40	42.8
iii)	15	30	32.1
iv)	10	20	21.4

8.2.3 *Atomic Absorption Operating Conditions*

Use a silicon hollow-cathode lamp and set the monochromator at 251.6 nm. Use a nitrous oxide/ acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

8.2.4 *Calculation*

Read out absorbance values for the blank solution (**8.2.2**), standard solutions, and sample solution (**7.2.4**). Determine percent SiO_2 as follows:

$$SiO_2\text{, percent} = [(C - B)/(A - B)] \times N \times 1.07 \quad \ldots\ldots(3)$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Silicon, in ppm, in standard.

NOTE — In all the calculations/determinations, the Atomic Absorption Spectrometer may be calibrated to read out "percent concentration" directly.

8.3 Aluminum Oxide (Al_2O_3)

8.3.1 *Preparation of Standards*

Prepare standard solutions containing 10 to 30 ppm aluminum and dilute with blank solution (**8.2.2**).

8.3.2 *Atomic Absorption Operating Conditions*

Use an aluminum hollow-cathode lamp and set the monochromator at 309.2 nm. Use a nitrous oxide-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

8.3.3 *Calculation*

Read out absorbance values for the blank solution (**8.2.2**), standard solutions (**7.2.4**), and sample solution. Determine percent alumina as follows:

$$Al_2O_3\text{, percent} = [(C - B)/(A - B)] \times N \times 0.94 \ \ldots\ldots(4)$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Aluminium, in ppm, in standard.

8.4 Iron Oxide (Fe_2O_3)

8.4.1 *Preparation of Standards*

Prepare standard solutions containing 2 to 10 ppm iron and 10 ml of lanthanum solution (**5.4.4**) per 100 ml. Dilute with blank solution (**8.4.2**).

8.4.2 *Blank Solution*

Prepare 0.5g/l of lithium tetra borate solution in solvent acid.

8.4.3 *Atomic Absorption Operating Conditions*

Use an iron hollow-cathode lamp and set the monochromator at 248.3 nm. Use an air-acetylene flame of stoichiometric composition. Adjust other instrument parameters to optimum for the particular equipment used.

8.4.4 *Calculation*

Read the absorbance values for the blank solution (**8.4.2**), the standard solutions, and the sample solution (**7.2.5**). Determine percent Fe_2O_3 as follows:

$$Fe_2O_3\text{, percent} = [(C - B)/(A - B)] \times N \times 2.86 \text{ ……..(5)}$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Iron, in ppm, in standard.

8.5 Calcium Oxide (CaO)

8.5.1 *Preparation of Standards*

Prepare standard solutions containing 1 to 10 ppm calcium and 10 ml of lanthanum solution (**5.4.4**) per 100 ml. Dilute with the blank solution (**8.4.2)**.

8.5.2 *Atomic Absorption Operating Conditions*

Use a calcium hallow-cathode lamp and set the monochromator at 422.7 nm. Use an air-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

8.5.3 *Calculation*

Read the absorbance values for the blank solution (**8.4.2**), the standard solutions, and the sample solution (**7.2.5**). Determine percent CaO as follows:

$$CaO\text{, percent} = [(C - B)/(A - B)] \times N \times 2.80 \text{ …....(6)}$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Clacium, in ppm, in standard.

8.6 Magnesium Oxide (MgO)

8.6.1 *Preparation of Standards*

Prepare standard solutions containing 0.5 to 2 ppm magnesium and 10 ml of lanthanum solution (**5.4.4**) per 100 ml. Dilute with blank solution (**8.4.2)**.

8.6.2 *Atomic Absorption Operating Conditions*

Use a magnesium hollow-cathode lamp and set the monochromator at 285.1 nm. Use an air-acetylene flame of stoichiometric composition. Adjust other instrument parameters to optimum for the particular equipment used.

8.6.3 *Calculation*

Read the absorbance values for the blank solution (**8.4.2**), the standard solutions, and the sample solution (**7.2.5**). Determine percent MgO as follows:

$$MgO\text{, percent} = [(C - B)/(A - B)] \times N \times 3.32 \text{ ……..(7)}$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Magnesium, in ppm, in standard.

8.7 Sodium Oxide (Na_2O)

8.7.1 *Preparation of Standards*

Prepare standard solutions containing 0.5 to 2 ppm sodium and 10 ml of lanthanum solution (**5.4.4**) per 100 ml. Dilute with blank solution (**8.4.2**).

8.7.2 *Atomic Absorption Operating Conditions*

Use a sodium hollow-cathode lamp and set the monochromator at 589.0 nm. Use an air-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

8.7.3 *Calculation*

Read the absorbance values for the blank solution (**8.4.2**), the standard solutions, and the sample solution (**7.2.5**). Determine percent Na_2O as follows:

$$Na_2O\text{, percent} = [(C - B)/(A - B)] \times N \times 2.70 \text{ ……..(8)}$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Sodium, in ppm, in standard.

NOTE — Alternatively, sodium may be determined by flame emission.

8.8 Potassium Oxide (K_2O)

8.8.1 *Standard Preparation*

Prepare standard solutions containing 0.5 to 2 ppm potassium and 10 ml of lanthanum solution (**5.4.4**) per 100 ml. Dilute with blank solution (**8.4.2**).

8.8.2 *Atomic Absorption Operating Conditions*

Use a potassium hollow-cathode lamp and set the monochromator at 766.5 nm. Use an air-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

8.8.3 *Calculation*

Read the absorbance values for the blank solution (**8.4.2**), the standard solution, and the sample solution (**7.2.5**). Determine percent K_2O as follows:

$$K_2O\text{, percent} = [(C - B)/(B - A)] \times N \times 2.41 \quad \text{.......(9)}$$

Where,

A = Absorbance of standard nearest C;

B = Absorbance of blank;

C = Absorbance of sample; and

N = Potassium, in ppm, in standard.

NOTES:

1 Alternatively, potassium may be determined by flame emission.

2 It may be found convenient to combine the standards for iron, calcium, magnesium, sodium, and potassium in one solution.

8.9 Titanium Dioxide (TiO_2)

8.9.1 *Standard Preparation*

Prepare a standard solution containing 3 ppm titanium and 5 g/l of lithium tetraborate solution dissolved in solvent acid. If required, successive dilutions may be made with the blank solution (**8.9.2**).

8.9.2 *Blank Solution*

Prepare a solution containing 5 g/l of lithium tetraborate solution dissolved in solvent acid.

8.9.3 *Sample Solution*

Use the original sample solution as prepared in **7.2.3**.

8.9.4 *Atomic Absorption Operating Conditions*

Use a titanium hollow-cathode lamp and set the monochromator at 364.3 nm. Use a nitrous oxide-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment use.

8.9.5 *Calculation*

Read the absorbance for the blank solution, the standard solution (**8.9.2**) and the sample solution (**8.9.3**). Determine percent TiO_2 as follows:

$$TiO_2\text{, percent} = [(C - B)/(A - B)] \times 1.00 \quad \text{.........(10)}$$

Where,

A = Absorbance of standard;

B = Absorbance of blank; and

C = Absorbance of sample.

9 REPORT

9.1 Report the percentages for the elemental oxides in the ash as specified in **8.2.5**, **8.3.3**, **8.4.4**, **8.5.3**, **8.6.3**, **8.7.3**, **8.8.3** and **8.9.5**.

9.2 Report the method used for concurrent moisture and ash determination, if applicable.

10 PRECISION AND BIAS

10.1 Precision

The precision of this test method is in accordance with Table 2.

10.1.1 *Repeatability Limit (r)*

The value below which the absolute difference between two test results of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 percent.

10.1.2 *Reproducibility Limit (R)*

The value below which the absolute difference between two test results, carried out in different laboratories using samples taken at random from a single quantity of material that is as nearly homogeneous as possible, may be expected to occur with a probability of approximately 95 percent.

10.2 Bias

Analysis of ash by flame Atomic Absorption Spectroscopic techniques is empirical and its approach to accuracy is dependent upon the accuracy of the reference materials to be used for calibration and compliance to this test method.

10.3 The Concentrations ranges and limits for repeatability and reproducibility for major elemental oxides in coal and coke ash are given in Table 2.

Table 2 Concentrations Ranges and Limits for Repeatability and Reproducibility for Major Elemental Oxides in Coal and Coke Ash

(*Clause* 10.3)

Sl No.	Elemental Oxide	Concentration Range, percent	Repeatability Limit (*r*)	Reproducibility Limit (*R*)
(1)	(2)	(3)	(4)	(5)
i)	SiO_2	33.14 – 57.73	$0.33+0.04\bar{x}$ ¹⁾	$-1.21 + 0.14\bar{x}$
ii)	Al_2O_3	11.32 – 31.83	0.80	$-0.24 + 0.09\bar{x}$
iii)	Fe_2O_3	2.93 – 41.11	$0.01 + 0.06\bar{x}$	$0.36 + 0.06\bar{x}$
iv)	MgO	0.45 – 7.12	$0.04 + 0.03\bar{x}$	$0.04 + 0.09\bar{x}$
v)	CaO	1.46 – 22.19	$0.21 + 0.04\bar{x}$	$0.15 + 0.11\bar{x}$
vi)	TiO_2	0.57 – 1.44	0.13	0.19
vii)	K_2O	0.45 – 2.99	$0.03 + 0.03\bar{x}$	$0.05 + 0.05\bar{x}$
viii)	Na_2O	0.14 – 7.15	$0.06 + 0.03\bar{x}$	$0.04 + 0.18\bar{x}$

1) Where $\bar{x}$ is the average of two single test results.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Solid Mineral Fuels and Solid Biofuels Sectional Committee PCD 07

Organization	*Representative(s)*
CSIR Central Institute for Mining and Fuel Research (CIMFR), Dhanbad	Dr Pradeep Kumar Singh (***Chairman***) Shri T. B. Das (*Principal*)
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Amendments Issued Since Publication

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